Synthesis and Characterization of Lithium Lanthanum Zirconate Solid State Electrolyte for Li - Ion Thin Film Batteries

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

by

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I hereby declare that the short synopsis entitled **"Synthesis and Characterization of Lithium Lanthanum Zirconate Solid State Electrolyte for Li ion thin film batteries"** being submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in the Department of Sciences (Physics) under the School of Sciences of Manav Rachna University, Faridabad during the academic year 2024-2025.

It is the work that is expected to be carried out under the supervision of Dr. Moditma and co-supervision of Dr. Jitendra Pal Singh and Dr. Aditya Sharma.

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ABSTRACT

A conventional lithium-ion battery employs hazardous, flammable electrolytes that contain organic solvents and lithium salts. Solid state electrolytes are usually employed for resolving these kinds of issues. The synthesis of garnet type Lithium Lanthanum Zirconium Oxide (LLZO) as a solid state electrolyte thin film using radio frequency (RF) sputtering and their characterization using X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Rutherford backscattering (RBS) are the subjects of this synopsis. The families of garnet materials exhibit significant electro-chemical performance along with the size reduction of device. But this material still faces challenges such as composition control, poor crystallinity, low ionic conductivity, interface stability and dendrite growth. LLZO exists in two phases i.e. tetragonal and cubic but to stabilize it in cubic phase is quite challenging. This study therefore aims at stabilizing cubic phase LLZO, with the required composition and crystalline structure.

Key words: Solid State Electrolyte, XRD, SEM, Garnet structure, LLZO

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1. Introduction

1.1 Battery

Since the invention of electricity as a source of energy, the use of lightweight electronics has increased significantly and batteries serve as important power source. Energy from chemicals that takes the form of electricity can be stored in batteries. An electrochemical reduction and oxidation reaction occurs at each battery electrode to provide electrochemical energy. Batteries are made up of a number of electro - chemical cells which are combined in series / parallel and offer the desired capacity and voltage performance. There are two categories of batteries: primary and secondary. While the electrochemical processes in secondary batteries can be reversible, in primary batteries, once chemical energy is converted to electrical energy, there is no way to undo the process. An external electric source is used to recharge secondary batteries. To recharge a battery; it is mandatory to have an effective electrolyte, two electrodes (i.e. +ve and –ve) and a current collector. The positive and negative electrodes inside and the current collectors outside are connected by an ionically conductive material known as the electrolyte. A battery's general schematic is displayed in Figure 1.

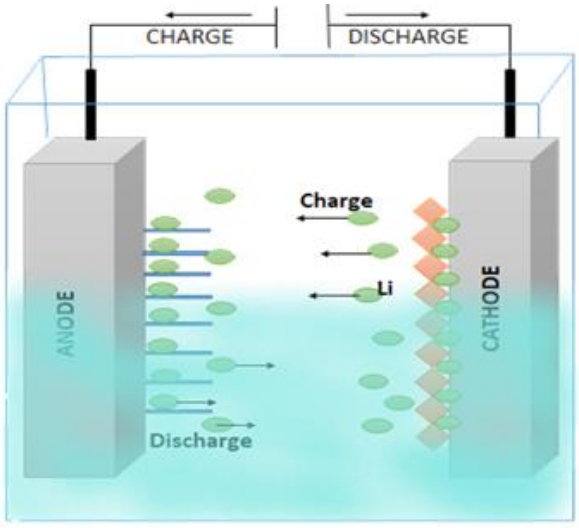

Figure 1 General schematic of battery.

1.2 Lithium – ion battery

Rechargeable Li-ion batteries are heavily utilized in daily lives such as mobile phones, electric car and portable devices like cell phones, tablets and portable computers. An organic electrolyte made up of lithium salt and the positive ve electrodes made of lithiated transition metal oxides serves as the separator in a conventional Li-ion battery. Li ions are able to transfer via electrolyte between the two electrodes. A lithium ion with an electron attached exits the negative electrode during discharge and enters the electrolyte in an ionic form.

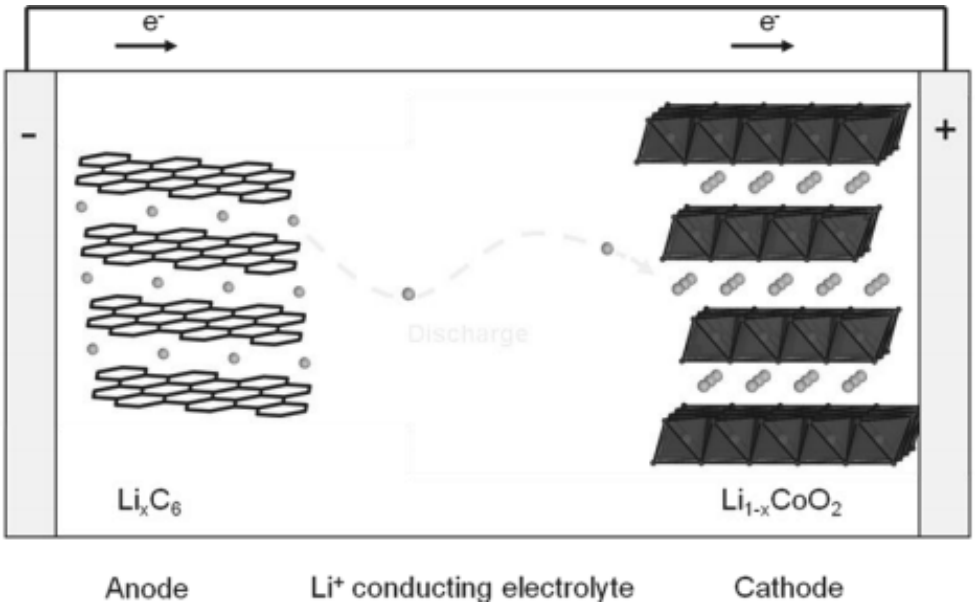

Figure 2: Representation of typical Li-ion battery

If B represents the species (Li) that carries charges, in the case of Li - ion batteries. (A) and (C) are the electrodes, respectively, then, as the discharge continues, B moves from electrode A to electrode C while simultaneously transferring electrons through the external circuit. The following equations can be used to express this:

$AB \rightleftarrows A + Bn+ + ne-$ (oxidation)

$C + Bn+ + ne- \rightleftarrows BC$ (reduction)

$AB + C \rightleftarrows A + Bn+ + ne- + C \rightleftarrows A + BC$ (oxidation - reduction reaction)

ΔG = - nEF

Where, The Gibb's free energy differential between the two electrodes is represented by the ΔG, **E** is the potential difference between the two electrodes, $\boldsymbol{n}$ denotes the number of +ve charges of the ions carry and **F** represents the Faraday constant.

1.3 Solid state lithium rechargeable thin film batteries

Recent years have seen rapid progress in the development of thermally and chemically strong, high performance rapid Li ion conductors for use in all solid state batteries [1]. By substituting the currently employed organic fluid electrolytes, which have protection drawbacks such as high toxicity, flammability, a narrow frame for chemical stability performance, solid state electrolytes (SSE's) are being integrated into Li+ batteries, increasing their reproducibility and safe uses [2-4]. Additionally, SSE's may prevent the growth of lithium dendrites, exhibit greater thermal stability, and permit the stacking of numerous cells for higher voltage devices [5-7]. There are 3 kinds of SSE's are being studied right now. Polymers are the first, while inorganic ceramics are the second [8-10]. The last category has been divided into garnet-types, sulfide-types, perovskite–type, sodium superionic conductor (NASICON)–type, Lithium superionic conductor (LISICON) - type materials [11-14]. The third is an integrated electrolyte that combines both inorganic ceramic and polymer [15-16]. The NASICON type electrolytes face challenges with their fast capacity decay and low coulombic efficiencies [17].

1.4 Garnet structured LLZO as a SSE

For batteries, one of the notable and promising SSEs is garnet electrolyte, which has benefits in terms of safe use, high-temperature stability, electro-chemical stability and high energy density [18-21]. LLZO has a garnet-like structure, which is made up of interconnected dodecahedra, octahedra, and tetrahedra. Each of these shapes has an oxygen atom at its corners, as shown in Figure 2. [22]. At ambient temperature, such garnet structured SSE's have moderate Li+ conductivity of 10^{-3} S/cm (conventional fluid electrolytes exhibit ionic conductivity of 10^{-2} S/cm) [23],[24], [25],[26].

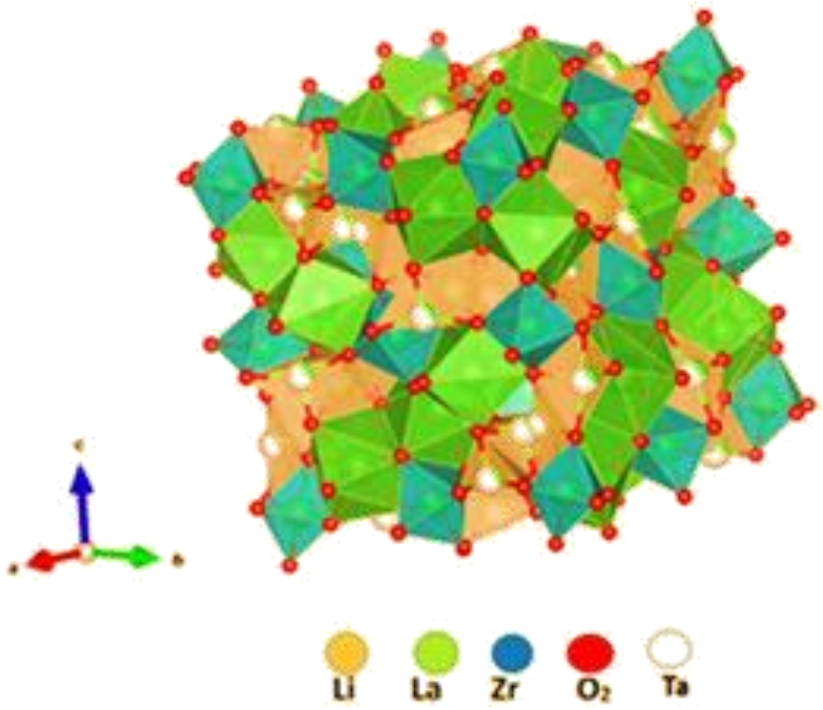

Figure 3 Garnet type LLZO (Vesta produced drawing)

The formula $Li_7A_3B_2O_{12}$ is commonly encountered in garnet-based structures. Here lithium ions are present at the octahedral and tetrahedral sites, A belongs to the 8 fold co-ordination sites and B to the 6 fold co-ordination sites. Tetragonal structure of LLZO belongs to *I41/acd* space group with cell parameters ($c \neq b = a$; $\alpha = \beta = \gamma = 90°$) on the other hand cubic structure belongs to *Ia3d* space group with lattice parameters ($a = b = c$; $\alpha = \beta = \gamma = 90°$). Tetragonal structure is thermodynamically stable at room temperature Compared to the garnet-type cubic structure in *Ia3d*, this structure has a lithium-ion conductivity that is roughly 2 orders of magnitude lower [27]. To stabilize the cubic structure at room temperature, aliovalent doping could be used. The Doping of the Li - position with using the elements or atoms are positioned at - Ga, Nb, Ta, W, Al, Zn and Fe can increase the ionic conductivity of lithium-ion batteries [28-31]. In tetragonal phase of LLZO, lithium ions are located in the octahedral (16f and 32g) and tetrahedral (8a) sites. However, in the cubic phase, lithium ions occupy only the octahedral (96h) and tetrahedral (24d) sites. This proves beyond doubt that there are more interstitial sites in cubic LLZO for Li ion migration [27]. Due to its strong overall performance, including improved Li+ conductivity and increased

electrochemical window (>5 V), and highly stable in air, the oxide containing SSE $Li_7La_3Zr_2O_{12}$ (LLZO) is regarded as a promising SSE material [32].

2. Literature Review

2.1 The Evolution of Lithium Rechargeable Thin Film Batteries

Whittingham initiated an experiment in 1972 with $LiClO_4$ in di-oxolane serves as the electrolyte, with TiS_2 serving as cathodic electrode and Lithium metal serving as anodic electrode. TiS_2 was the most effective intercalation substance during that time, possessing an extremely reasonable layered structure [33]. It was soon found that one of the disadvantages of the lithium-metal/liquid electrolyte combination, which raises explosive issues, is uneven lithium development as metal was replaced after each subsequent dis-charge and re-charge cycle. Meanwhile, major breakthroughs in intercalation materials had occurred as a result of understanding the oxides, in addition to their initial attention in denser chalcogenides, were providing larger capacities and voltages [34]. Li_xMO_2 was found as a cathode material by K. Mizushima and his colleagues. Various other methods were explored to circumvent the safety issues related to the use of lithium metal, such as adjusting the anode electrodes or the electrolyte [35]. $C/LiCoO_2$ (LCO) batteries, which were successfully commercialized in 1994 and swanked an exceptional energy density of 120 –150 W h/kg (2–3 times more than standard Ni-Cd batteries) was regarded the top performance portable power devices [36].$LiCoO_2$ has now becoming the ultimate common and extensively utilized cathodic solid. J. B. Bates in 1992 preparation of solid state electrolyte thin films of Lithium phosphorus oxynitride (Li-PON) was another breakthrough moment [37]. Currently, LiPON is widely acknowledged as a typical solid electrolyte utilized in LIBs. Following that, J. Bates et al. produced thin films using glassy electrolyte LiPON once more in the year 2000. Here, with crystalline $LiCoO_2$ cathode, up to 30% capacity in between 4.2V to 3V at a discharge current of $10mAcm^{-2}$ was achieved. [38] In 2007, 3D integrated LIBs ($LiCoO_2$-LiPON-Si) were proposed to boost LIBs' energy density [39]. Cras et al. (2015) reported micro scale LIBs ($Li_{1.2}TiO_{0.5}S_{2.1}$-Li-PON-Si) with a cycled capacity of about 38 $\mu Ahcm^{-2}/\mu m$ over 740 cycles [40]. STM microelectronics in the year 2017 suggested LIBs of $LiCoO_2$-LiPON-Li with the minimum power of 3.9 volt and an internal capability of capacity 1 m A h [41]. Garbayo and his co - workers (2018) employed Pulsed Laser Deposition (PLD) to deposit the

$Li_7La_3Zr_2O_{12}$ (LLZO) thin film and obtained potential solid state electrolyte thin films for upcoming LIBs through varying the deposition parameters such as temperature to improve ionic conductivity [42]. In the year 2020, TDK Electronics announced a 100 μAh all-ceramic structural lithium rechargeable battery with an initial voltage of 1.5 V. Aside from those mentioned breakthroughs, other businesses, including Infinite Power Solutions, Cymbet and EnFilm, have begun to commercialize LIBs for powering microelectronic devices. Companies like Infinite Power Solutions, Cymbet, and EnFilm have now started to commercialize lithium-ion batteries (LIBs) for powering microelectronic devices.

3. Description of broad area

The adaptation of solid state electrolytes rather than liquid electrolytes is focused on improving the various parameters such as: safety, power considerations, energy density, portability and dendrite formations. SSEs like NASICON, LISICON, Perovskites, and Argyrodrites have shown good properties but suffer from low stability and interfacial issues. Garnet-structured LLZO stands out as a superior SSE due to its high electrochemical and interfacial stability, as well as moisture resistance. While LLZO exists in both tetragonal and cubic phases, the cubic phase exhibits higher stability and ionic conductivity of 10^{-4} Scm^{-1}, making it a promising candidate for thin film rechargeable batteries. LLZO based solid state electrolytes hold great potential for high energy density Li ion batteries with enhanced safety. Many fundamental concerns however must be addressed before they can be used in practical applications:

(1) Low ionic conductivity of solid state electrolytes

(2) Crystallization of LLZO in cubic phase

(3) High interface resistance

(4) Unwanted dendrite growth

Here in this study, we aim to address some of these challenges.

4. Objectives of the study

This research work aims to address the following goals:

- ✓ Growth of LLZO thin films on Si and Glass substrates using radio frequency (RF) sputtering.
- ✓ Stabilization of Cubic Phase of LLZO at suitable annealing temperature
- ✓ Composition and thickness optimization
- ✓ Optimization of ionic conductivity for better device performance

5. Methodology to be adopted

5.1 Preparation of LLZO thin films

Thin films of LLZO will be deposited by using RF sputtering technique. This involves optimization of various parameters such as RF power, base pressure, substrate temperature, Ar gas flow rate, as well as the deposition time. Post synthesis, the prepared films will be annealed at different temperatures, by varying the heating and cooling rates in a muffle furnace, for enhanced sample properties.

5.2 Characterization and Analysis

The prepared films will be characterized for their structural, morphological and electrochemical properties using the following techniques:

- ❖ Morphology and surface roughness determination using Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM)
- ❖ Structural analysis using X- ray Diffraction (XRD).
- ❖ Fim thickness and composition estimation using Rutherford Backscattering Spectrometry (RBS)
- ❖ Understanding the local electronic structure using X-ray Absorption Spectroscopy (XAS)
- ❖ Ionic conductivity measurements

6. Expected outcomes of the research

This research work aims at optimizing the crystalline phase and composition of LLZO thin films, to be used as solid state electrolytes for thin film rechargeable battery applications. This will be useful for obtaining low cost, portable, solid state batteries with enhanced electrochemical properties, to be used in electric vehicles, biomedical devices, portable electronic devices, and much more.

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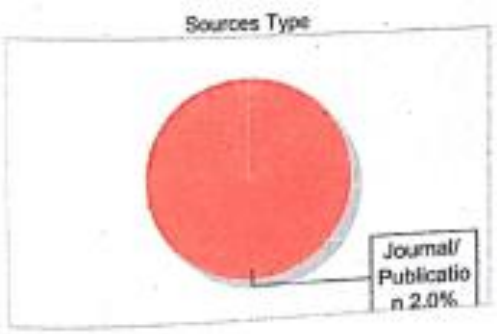

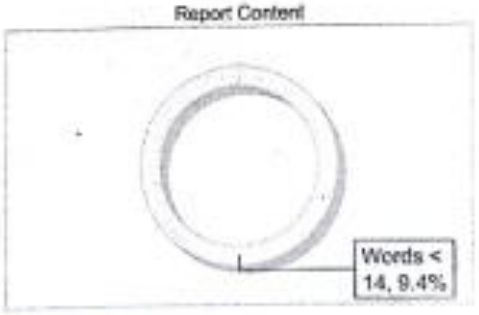

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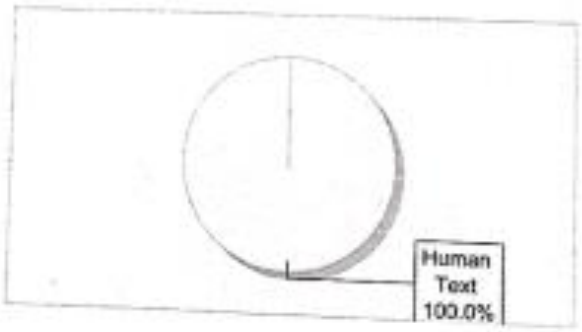
Submission Information

Author Name	Anjali
Title	Synthesis and Characterization of Lithium Lanth..
Paper/Submission ID	2320916
Submitted By	yogeshkumarmalik.library@mru.edu.in
Submission Date	2024-09-17 15:53:10
Total Pages	11
Document type	Synopsis

Result Information

AI Text: **0 %**



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